MODIFICATION OF AN ULTRA-HIGH-VACUUM SCANNING TUNNELING MICROSCOPE FOR SILICON NANOSTRUCTURE FABRICATION

BY

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THESIS

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Abstract

In this thesis, two major modifications to an ultra-high-vacuum scanning tunneling microscope system are described: an update to the cooling plate structure for more effective cooling of the dipstick and sample holder, and the installation of a capillary doser for concentrating the precursor gas to the tip-sample junction during silicon nanostructure growth. The updated cooling plate is able to shorten the total sample preparation time from 6 hours to 3 hours. The capillary doser lowers the base pressure during the silicon growth experiment by two orders of magnitude. The system operation after the system modification was tested. The system is now ready for subsequent silicon nanostructure growth using disilane gas.

To my parents for always believing in me.

To my husband Feng for his love and support.

Acknowledgments

I would like to express my sincere gratitude to my adviser, Professor Joseph Lyding for, the opportunity to pursue my graduate degree in his research group, and for his guidance and encouragement during the course of my research. I would like to acknowledge valuable discussions with my colleagues in the Lyding group: Scott Schmucker, Wei Ye, Justin Koepke, Pam Martin, Josh Wood, Kevin He, Greg Scott, Sumit Ashtekar and Jae Won Do. I would like to thank Scott Robinson and Cate Wallace from the Imaging Technology Group at the Beckman Institute for their technical support with the TEM, and Craig Zeilenga from the ECE machine shop for his help in building various parts in the STM system. Finally, I would like to thank all of my family and friends for their loving support. This work was supported in part by the Defense Advanced Research Projects Agency (DARPA) and Office of Naval Research (ONR).

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Chapter 1: Introduction

1.1 Background

The scanning tunneling microscope (STM) is a powerful instrument that can provide three-dimensional images of a sample surface at the atomic level. It was developed in 1981 by Gerd Binnig and Heinrich Rohrer, who received half of the 1986 Nobel Prize in Physics for this important invention. Binnig and Rohrer describe the development of STM in [1].

STM uses feedback control to monitor the tunneling current between the sample surface and a metallic probe, and adjusts the sample-probe distance to maintain a constant current. The change in the tunneling current can be translated into a topographic profile, with the ability to image individual atoms. In addition, STM is capable of manipulating single atoms [2] and collecting data on the electronic properties of the sample through scanning tunneling spectroscopy (STS) on individual atoms [1, 3]. The ability to image and modify the sample surface with atomic resolution *in situ* makes STM an important instrument in research.

All the work presented in this thesis was collected using a room temperature ultra-high-vacuum scanning tunneling microscope (UHV-STM) custom designed by Professor Joe Lyding and built by Dr. Matthew Sztelle. The UHV system consists of various components which work together to ensure the operation of the STM. As part of this thesis, modifications to these components have been made to improve the overall operation of the system.

1.2 Thesis Overview

Building upon a foundation requires understanding of that foundation. Therefore, Chapter 2 will begin with an overview of STM operating principals, followed by a brief discussion of the Lyding STM scanner and the ultra-high-vacuum chamber designed to accommodate the scanner. Typical silicon sample preparation steps and tip fabrication techniques will also be discussed.

Chapter 3 will discuss the two major modifications to the STM system described in the thesis: the cooling plate update and the capillary doser installation. Due to the nature of UHV, any system modification requires careful consideration and precise treatment.

The design and construction of the cooling plate and capillary doser will be discussed in detail. Furthermore, the effectiveness of these updates will be discussed. Tests of system operation after these modifications, along with STM images, will be presented.

Chapter 4 will conclude the thesis, and finally the appendix includes the drawings used for all the pieces in the assembly of the cooling plate.

Chapter 2: Experimental Setup and Methods

2.1 Scanning Tunneling Microscope Basics

The operation of an STM is based on the quantum tunneling of electrons between a metallic probe tip and a conducting or semiconducting sample. The vacuum gap between the sample and the tip acts as a potential barrier for the electrons. Classically, when the electron encounters a potential barrier higher than its energy, it will be deflected and will not pass through. However, on the quantum scale, the electron can be treated as a probability wave that decays exponentially when it encounters a finite potential barrier. Thus, if the potential barrier is narrow enough, there is a finite probability that the electron can tunnel to the other side, even though its energy is lower than that of the barrier (Figure 2.1).

Figure 2.2 shows how the concept is applied to the STM operation. When the STM tip is brought very close to the sample (usually ~ 1 nm), the electrons initially confined in the potential wells of the tip and sample have a finite probability of tunneling through the gap between them and entering the other potential well. Typically, the tip is grounded. Under negative sample bias, the electrons tunnel from the sample (filled states) to the tip; under positive sample bias, the electrons flow from the tip to the sample (empty state). The tunneling current I can be expressed as a function of sample voltage V, local density of states of the sample at the Fermi level $\rho_s(E_F)$, decay constant κ, the average sample-tip work function φ, and the tip-sample distance W. The complete expression is [4]

$$\begin{aligned} I &\propto V\,\rho_s(E_F)\,e^{-2\kappa W} \\ &\approx V\,\rho_s(E_F)\,e^{-1.025\sqrt{\phi}W} \end{aligned} \tag{2.1}$$

The most common mode of scanning is the constant current mode, which is the mode used for the work presented in this thesis unless otherwise stated. Figure 2.3 is a schematic of the STM operating in this mode. The tip is moved closer to or farther from the sample by a piezoelectric tube in the scanner. The real-time tunneling current is fed back to the control logic, and the voltages to the piezoelectric tube are adjusted to vary the tip-sample distance to maintain a constant tunneling current. The adjustments made to the piezoelectric tube are recorded and transformed into a topographic image of the surface, which can reach sub-angstrom resolution.

Since the tunneling current is a convolution of the tip and surface states, the STM

image provides both topographic and electronic information about the scanned surface. Unless the scanning surface is electronically uniform (in which case the displayed image should be a true representation of the surface topography), the image will be affected by the local density of states on the surface. For example, when scanning over a dangling bond (a missing hydrogen atom) on a H-passivated Si(100) surface, the resulting image would show a protrusion from the surface instead of a depression as we intuitively expect (Figure 2.4). This is because, at the dangling bond location, the clean silicon surface has a higher density of states, resulting in a higher tunneling current and causing the tip to move away from the surface. Thus we should keep in mind that an STM image is not a direct translation of the topographic profile of the scanning surface, and interpretation of STM images is important.

2.2 STM Scanner - The Lyding Design

The STM scanner developed by Lyding et al. [5] is a compact and rigid unit that fully compensates for thermal expansion or contraction. Figure 2.5 (a) is a CAD rendering of the Lyding microscope, and Figure 2.5 (b) is a photograph of the actual scanner used in this system. This design utilizes two concentric piezoelectric tubes. The inner tube is used for scanning and coarse translation of the tip, while the outer tube is used for thermal compensation and translation of the sample by an inertial translation scheme. To translate the sample toward the tip, a ramp voltage with a slow rising edge and a fast falling edge is applied to the outer piezoelectric tube. At the rising edge, the outer piezoelectric tube contracts and brings the sample and tip closer, while at the falling edge, the voltage is rapidly returned to its initial value, causing the tube to rapidly expand back to its initial position. In this process, the inertia of the sample holder prevents it from following the rapid motion of the rails. Thus, when the outer tube returns to its initial position, the sample holder has been translated toward the tip by one "step." By changing the amplitude and polarity of the ramp signal, the sample can be translated toward or away from the tip at different step sizes.

There are several advantages of this design. The concentric piezoelectric tubes provide low thermal drift (less than 2 Å/hour) and smooth sample and tip translation, and they eliminate the need for mechanical components such as springs, gears, or stepper mo-

tors that are known to result in considerable vibration sensitivity.

In the updated Lyding-design STM, invar replaces quartz as the base that holds the piezoelectric tubes, and as the rails which support the sample holder. Invar (64% Fe and 36% Ni) has a coefficient of thermal expansion 50 times lower than aluminum [6]. It is used to further reduce the thermal drift. In addition, the invar rails and the sample holder are coated with titanium nitride (TiN) (Replikote C, Richter Precision, East Petersburg PA), to reduce the coefficient of friction and to improve efficiency of sample translation [5].

The scanner in this system is the latest design of Professor Lyding. In addition to the standard features in the prototyped scanner mentioned earlier, the latest design has four additional electrical contacts to the sample holder. The standard scanner makes use of only two electrical contacts (upper rail and lower rail, Figure 2.5(a)) for sample biasing and heating. For most imaging applications, it is common to shunt these together, while during sample heating, a bias is applied across the two rails.

Figure 2.6 shows the newest scanner with the additional four rails. The rails are housed in a separate attachment, which is clamped to a modified invar tube. Like the standard rails, the additional rails are coated with TiN, which protects the rails and extends their lifetime. The additional rails provide the opportunity to run more sophisticated experiments with the STM. For example, thermocouple wires can be fed to a pair of the rails for real-time temperature measurement, and additional sample biasing can be achieved from the other pair of rails.

2.3 The Ultra-High-Vacuum System

Though STM can be operated under various pressure conditions, ranging from atmospheric, to high vacuum, to ultra-high-vacuum (UHV), lowering chamber pressure improves the resolution of images and produces more stable images. UHV reduces oxidation and contamination of the sample, and enables achieving atomic resolution on highly reactive clean metal and semiconductor surfaces. To operate an STM system under UHV, many supporting structures are required besides the STM itself. Figure 2.7 shows the actual UHV-STM system used in this work.

A loadlock is designed for sample introduction to and retrieval from the UHV

chambers without causing a vent to atmospheric pressure. A gate valve separates the loadlock from the UHV chamber. When a sample is loaded into the loadlock, the loadlock is first pumped down by a mechanical roughing pump to ~10^{-3} torr, and then by a turbomolecular pump to ~10^{-8} torr. The gate valve is then opened to transfer the sample into the UHV chamber. Linear translation manipulators (LTMs) are used to move sample/tip carriers within the system and exchange them between the loadlock and the UHV chamber.

The UHV chamber consists of three primary parts: the interchange (to exchange sample carrier between the two LTMs), the preparation chamber, and the main STM chamber.

Figure 2.8 shows the inside of the preparation chamber, where sample/tip preparation takes place. Three main parts can be seen in the figure: the dipstick (for sample/tip heating and biasing, and to provide rotational freedom), the cooling plate (as a heat exchanger for cooling the sample and the dipstick), and the cracking tungsten filament (to break hydrogen molecules into atomic hydrogen for H-passivation of silicon). It is worth mentioning that the cooling plate structure is unique to this system, and was recently updated to improve the cooling efficiency substantially. Further details can be found in Chapter 3.

Figure 2.9 is a photograph of the STM chamber. Electrical connections to the STM scanner are made from the top of the chamber. The scanner is located on an aluminum stage which is located at the bottom of the STM chamber. The stage is vibrationally isolated by long springs suspended from the top of the chamber. The pumping system (titanium sublimation pump (TSP) and ion pump) for maintaining UHV is connected to the chamber from underneath. Also attached to the chamber is the residual gas analyzer (RGA) used to monitor partial pressure of the background gases present. The newest addition to the system is a capillary doser installed to the STM chamber. The capillary doser will enable the base pressure in the system to be reduced by two orders of magnitude during the disilane experiment (more details in Chapter 3).

To bring the system into UHV from ambient pressure, a “bakeout” process is required. The chamber is wrapped by heating tapes and aluminum foil to achieve uniform heating of the chamber to 150 °C, while being pumped by a turbo-molecular pump. In the

early stage of the bakeout, the pressure will increase drastically due to the outgassing of impurities on the chamber walls. However, the pressure drops to ~10^{-8} torr over the course of approximately five days, as most of the desorbed gases are pumped away by the turbo-molecular pump. The system is then cooled down and the pressure drops further to below 10^{-10} torr, which is mostly accounted for by hydrogen.

2.4 System Operation

2.4.1 Sample Preparation

The samples used in this study are from boron-doped p-type Si(100) wafers. Sample preparation begins with cutting a small rectangular piece from the larger silicon wafer using a diamond scribe or wafer dicing saw. The sample size is usually 9 mm x 4 mm to fit into the sample holder. The sample is then degreased by sonicating in acetone and isopropyl alcohol to remove organic impurities and dust particulates on the sample. Metal contact to the sample should be avoided after the degreasing to prevent nickel contamination. Thus the sample will only be handled by Teflon tweezers.

Figure 2.5(b) shows a typical sample holder used to mount samples. The sample holder embodies a "bracket design," in which the bracket is an extension of the invar sidepieces. A plate pushes the sample into the bracket when the screw behind the plate is tightened. A spring is inserted between the screw and the plate to allow the sample to expand (while being heated) without cracking. The invar sidepieces are coated with TiN to prevent nickel from diffusing onto the sample. In all the experiments, 1 mil thick tantalum foils are used to sandwich the sample at its sides touching the sample holder. The tantalum foil forms a robust silicide contact with the sample and prevents direct contact between the sample and the sample holder.

With the sample properly mounted, the sample holder is first placed into the loadlock, and is then transferred via the LTMs to the dipstick located in the preparation chamber.

The sample degassing takes place on the dipstick, by passing current through the two sides of the sample holder. This degas step is essential to remove any physisorbed contaminants on the sample / sample holder which can interfere with the scanning surface. The typical degas temperature for silicon sample is 600 °C. The sample temperature is

measured through a window using an infrared pyrometer. During the initial stage of the degas, the pressure in the preparation chamber rises by two to three orders of magnitude due to the outgassing of the sample and sample holder. As the degas continues, the pressure eventually drops back to ~10^{-10} torr. The degas period is usually 8-10 hours, and is followed by hydrogen passivation of the sample.

The sample is first flashed at 1250 °C for 30 seconds to form a low resistance silicide contact with the tantalum foil (Section 2.4.2). Also, the flash removes any native oxide and silicon carbide, leaving a very reactive clean silicon surface. The monohydride layer is formed by heating the sample to 377 °C in a hydrogen environment for 10 min. A tungsten filament heated to about 1300 °C is brought close to the sample during the process. The filament cracks the relatively inert H_2 molecules into highly reactive atomic hydrogen which reacts with the clean silicon surface to form the monohydride (2x1 reconstruction) layer.

The sample is now ready to be transferred into the STM chamber for scanning and subsequent experiments.

2.4.2 Tip Preparation

As mentioned in the previous section, the STM image is a convolution of the tip and surface density of states. Therefore, good tips are essential for achieving high resolution STM images. A sharp tip with a small radius of curvature ensures that only the atoms at the tip apex will interact with the sample surface, and the tunneling current results from the interaction of the tip and a very small area on the sample. The tips used in this work are made from electrochemically etched tungsten wires. A drop-off method is adopted (Figure 2.10): a gold-plated ring holding a film of 3M NaOH solution is biased at a positive voltage (commonly ranging from 2.5 to 4.0 V) relative to the tungsten wire, which is held at the center of the ring. The portion of the wire under the solution will eventually drop off after all the tungsten is etched away at the tungsten-NaOH junction. The drop-off tip is rinsed in deionized water to remove any residual NaOH. Figure 2.11 shows the TEM image of a typical tungsten drop-off tip. The radius of curvature at the tip apex is ~10 nm.

Tips are then placed in tip holders, which will be resistively heated after mounting

on a tip heater/carrier. A tip heater can hold up to four tips. It enters the UHV chamber in the same way as the sample, and is placed onto the dipstick for tip heating before it is transferred to the STM chamber. The tip heating/degassing cycle is usually done by passing 10 A current through the tip heater over a period of 8-10 hours.

A typical STM image of a Si (100)-2x1 surface prepared using the above recipes for sample and tip preparation is shown in Figure 2.4. The image exhibits dimer row resolution. The atomically flat silicon surface with dimer row directions perpendicular to each other in adjacent terraces can be seen.

2.5 Figures

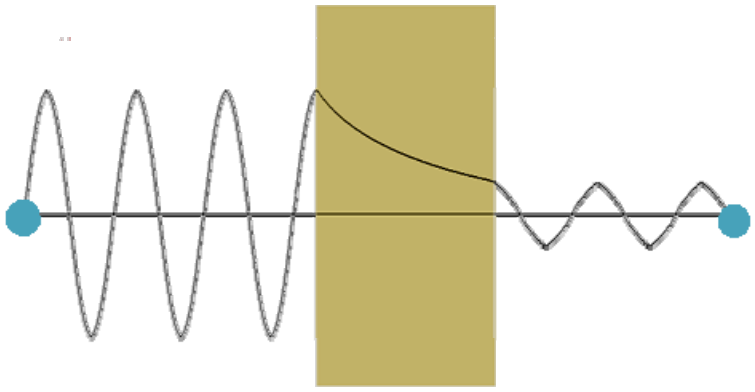

Figure 2.1: Quantum tunneling of an electron through a potential barrier higher than its energy. The electron wavefunction decays exponentially inside the barrier. With a potential barrier narrow enough, there is a finite probability that the electron can tunnel to the other side.

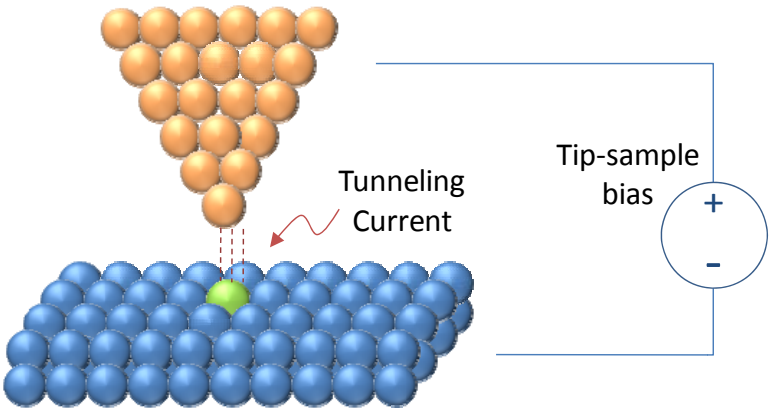

Figure 2.2: Illustration of tip-sample junction in STM. A voltage bias is applied over the sample and the tip. When the space between the two is very small (~1 nm), a tunneling current can flow through the junction.

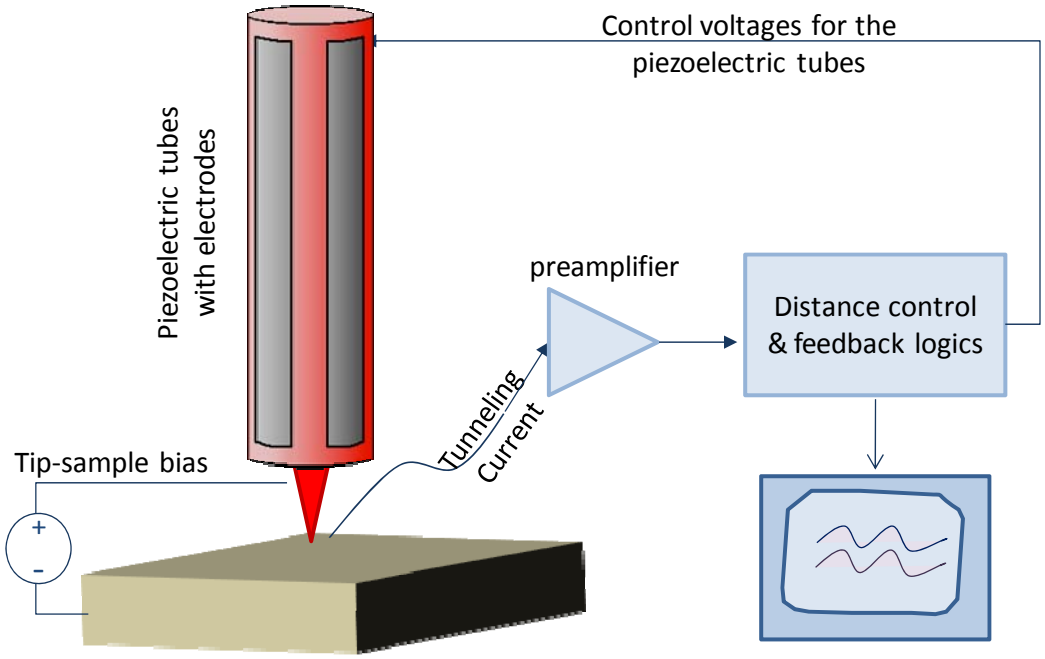

Figure 2.3: Schematics of STM operation. The tip is brought closer to or farther from the sample by a piezoelectric tube in the scanner. The real-time tunneling current is fed back to the control logic, and the voltages to the piezoelectric tube are adjusted to vary the tip-sample distance to maintain a constant tunneling current. The adjustments made to the piezoelectric tube are recorded by the computer and used to construct a topographic image of the surface.

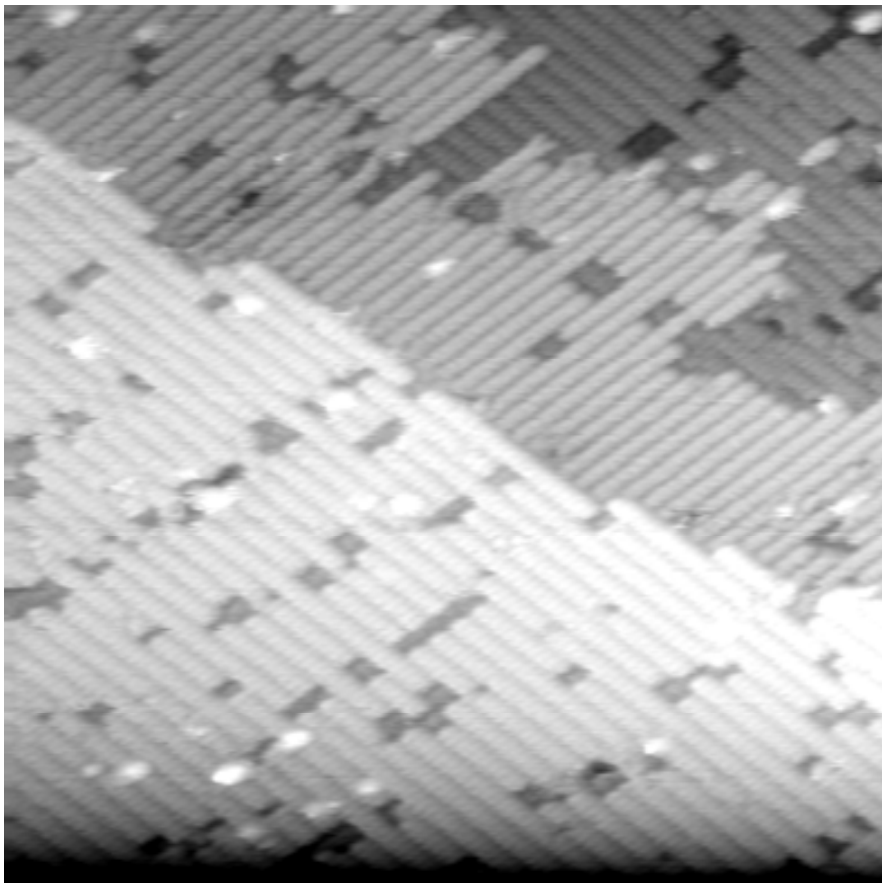

Figure 2.4: STM image of a H-passivated Si (100)-2x1 surface with dimer row resolution. Image size: 40 x 40 nm, scan condition: -2V, 0.1 nA. The flat silicon surface with dimer row directions perpendicular to each other in adjacent terraces can be seen. Bright spots represent dangling bonds, which appear to be surface protrusions due to high local density of states. Darker spots represent surface depressions (vacancies).

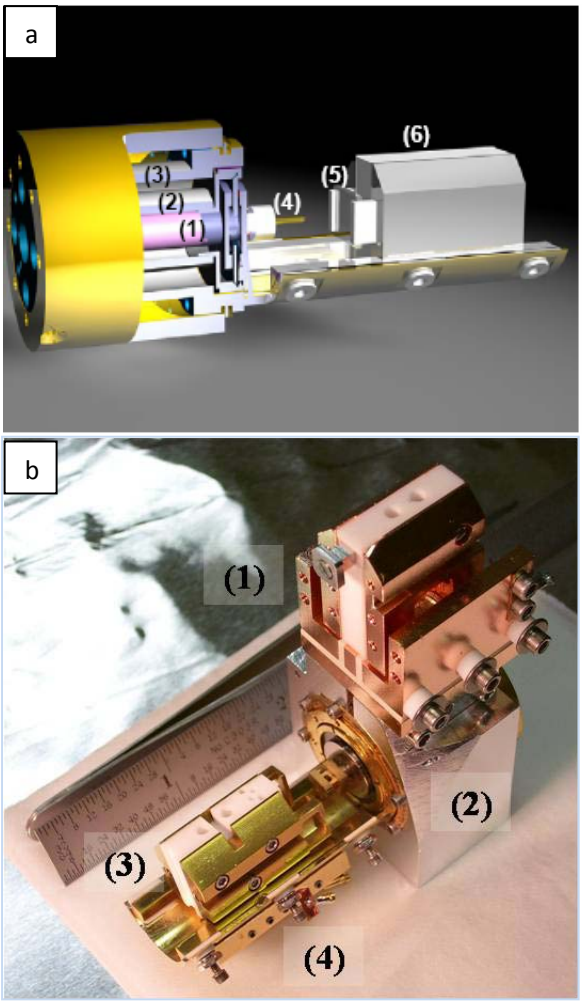

Figure 2.5: (a) CAD rendering of Lyding-design scanner and sample holder. A cross-section of the STM is shown to highlight the components of the scanner. (1) Tip translation assembly, (2) inner piezo tube, (3) outer piezo tube, (4) metallic tip, (5) sample, (6) sample holder. [CAD rendering courtesy of Joseph Lyding] **(b)** Photograph of microscope used in the system. (1) Molecular doser, (2) STM hidden by aluminum mounting block, (3) sample holder, (4) rails supporting the sample holder and enabling sample bias. [Courtesy of Dr. Matthew Sztelle]

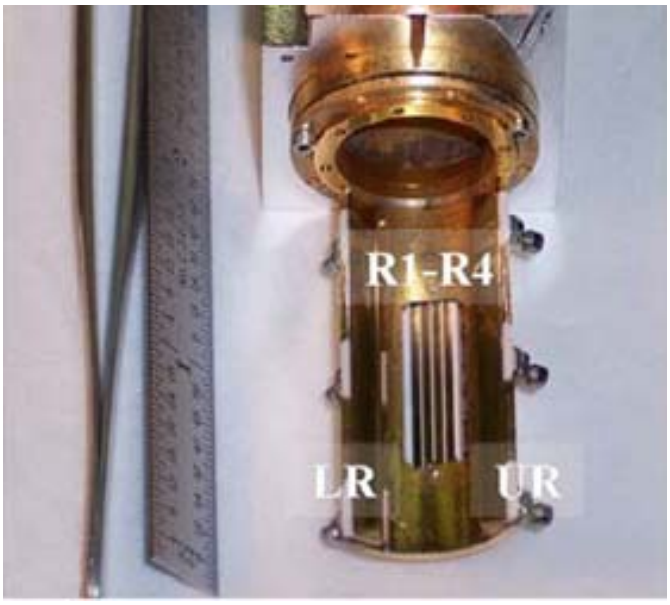

Figure 2.6: Photograph of the latest STM scanner with four more rails (R1-R4) in addition to the standard two rails (LR and UR). [Courtesy of Dr. Matthew Sztelle]

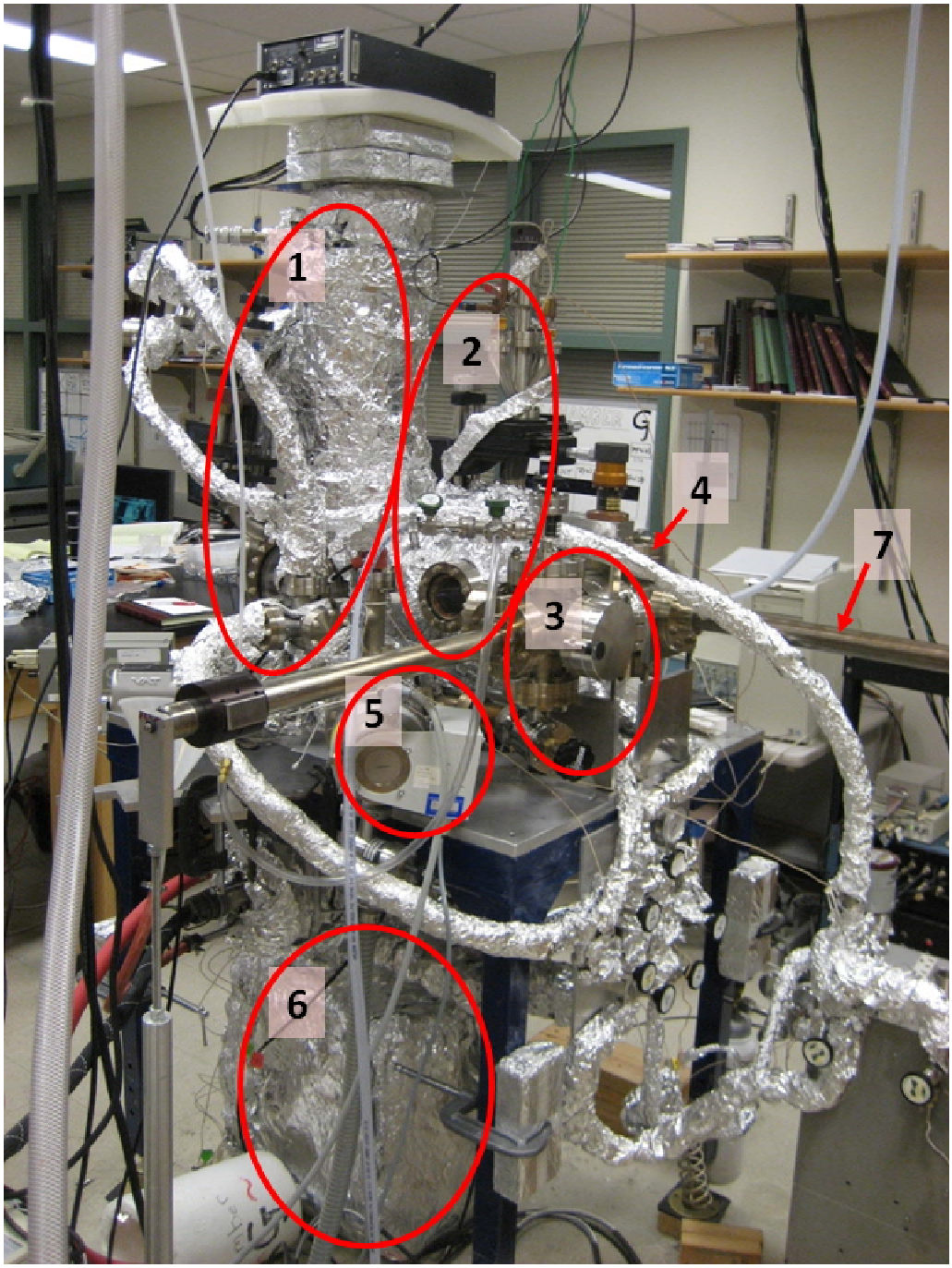

Figure 2.7: The STM system used in this work. (1) STM main chamber, (2) preparation chamber, (3) loadlock, (4) interchange, (5) turbomolecular pump, (6) ion pump, (7) linear translation manipulator, LTM.

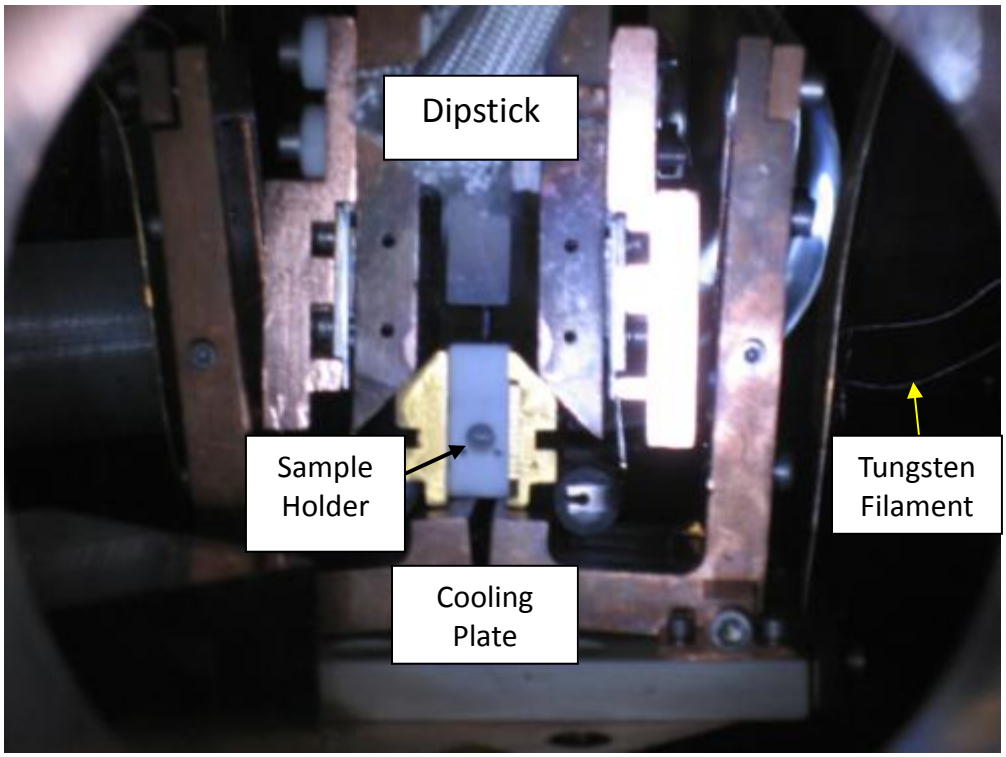

Figure 2.8: Photograph of the inside of the preparation chamber.

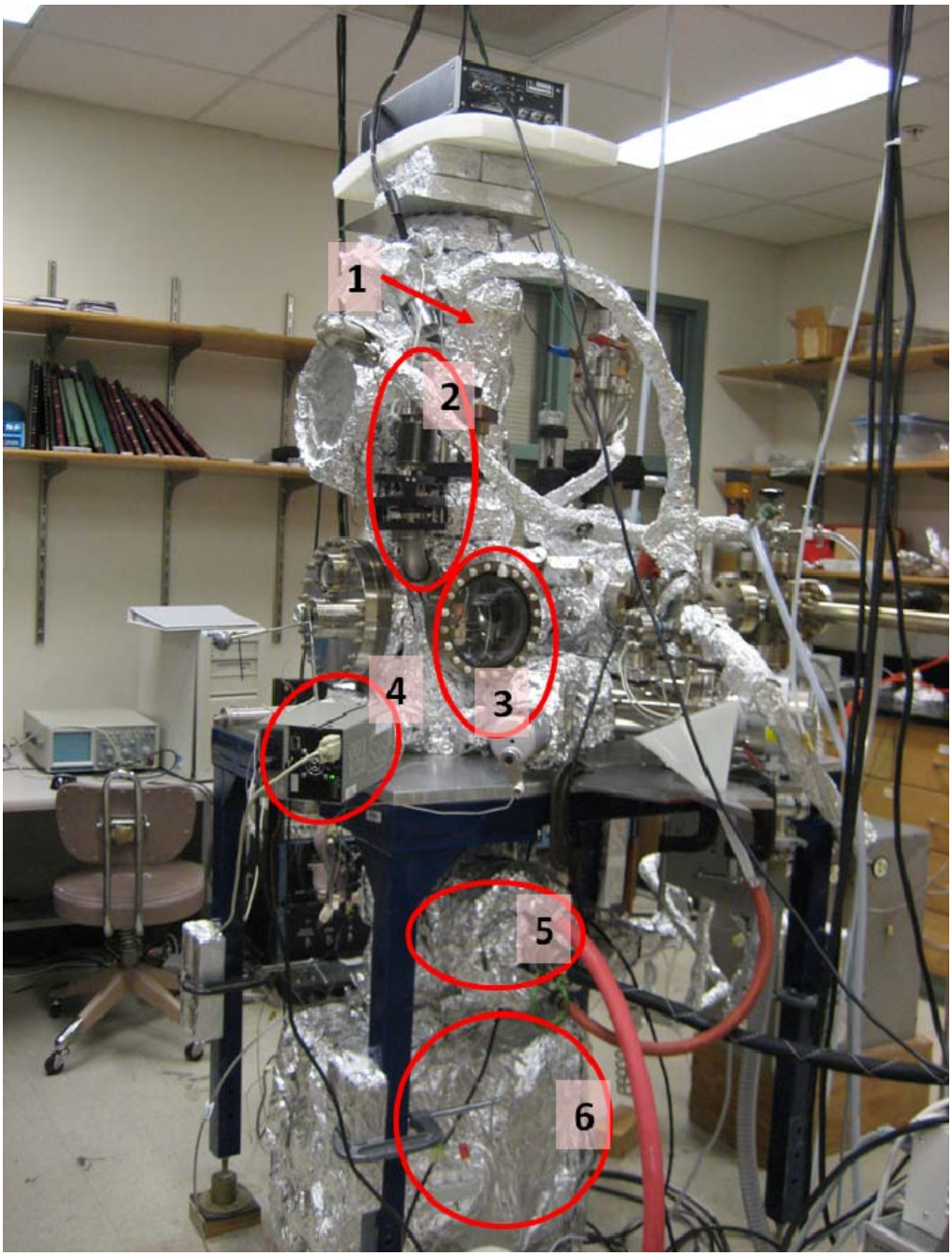

Figure 2.9: Photograph of the main STM chamber. (1) Housing of the springs for vibration isolation of the stage, (2) capillary doser manipulator, (3) STM scanner housing; (4) residual gas analyzer, (5) titanium sublimation pump, (6) ion pump.

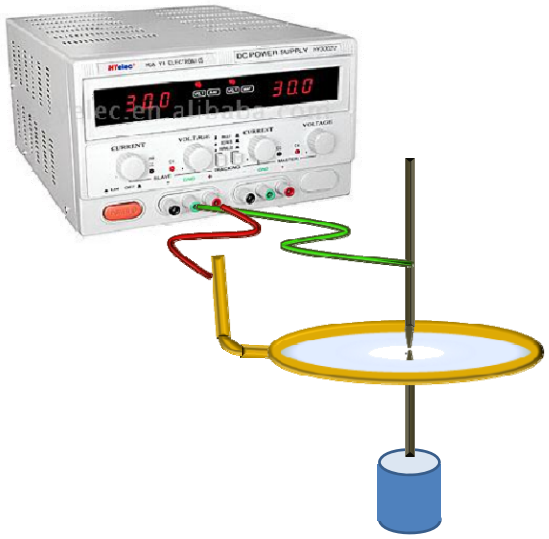

Figure 2.10: Tungsten tip etching setup. A gold ring holding a 3M NaOH solution is biased at a positive DC voltage relative to the tungsten wire, which is held at the center of the ring. The portion of the wire under the solution will eventually drop off after all the tungsten is etched away at the tungsten-NaOH junction.

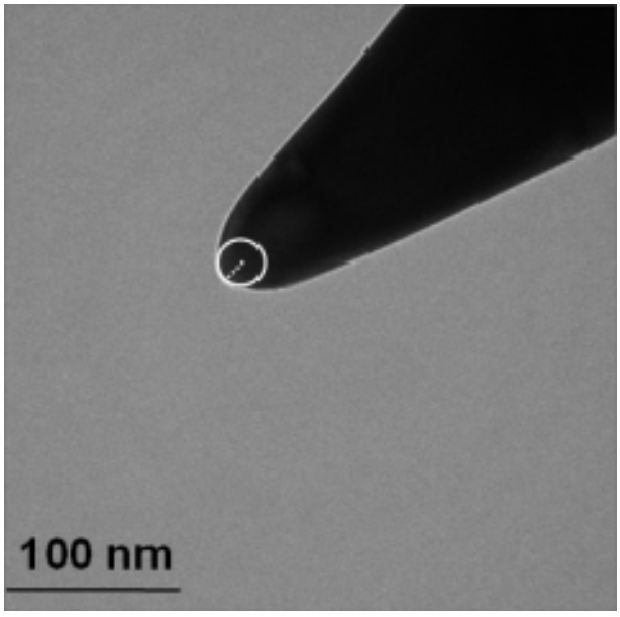

Figure 2.11: TEM image of a typical tungsten drop-off tip. The radius of curvature at the tip apex is ~10 nm.

Chapter 3: STM System Update

3.1 Cooling Plate Update

A cooling plate is a unique feature in the STM system used in this work. It is used to cool the dipstick and sample/tip carrier after high temperature processes (degas, flash or passivation). Cooling is an essential step in sample preparation. The sample and the dipstick need to be cooled to room temperature before the sample can be flashed to avoid contamination. In the process of sample preparation, cooling needs to be done at least three times: after sample degas (dipstick temperature $T_{DS} \approx 170$ °C), after the tungsten cracking filament degas ($T_{DS} \approx 75$ °C), and after the passivation process ($T_{DS} \approx 60$ °C). Out of the three cooling steps, the one after sample degas usually takes the longest. Overall, the speed of these cooling processes greatly affects the duration of sample preparation.

3.1.1 Original Cooling Plate

The original cooling plate design was a copper plate located at the bottom of the preparation chamber (Figure 3.1). The upper portion is made narrower so that the sample will be sitting on the narrower portion, while the rollers holding the sample holder can be fitted above the underlying layer. Nitrogen gas is passed through the cooling plate to keep it at a relatively low temperature with respect to the dipstick. Liquid nitrogen can also be passed through it, making the cooling faster. The copper plate rests on a support structure or "legs." It is made of copper blocks with Shapal-M sheets in between, as illustrated in Figure 3.1. A tantalum filament is sandwiched between the Shapal-M sheets as a heating source for cooling plate degas.

This design eliminates the use of a differential stage design in the dipstick as is used in all the other systems in our laboratory. In that case, the dipstick is cooled by an internal cooling pipe where nitrogen gas is passed through. The cooling plate design is more straightforward and should be effective in theory. However, while the MACOR center piece of the sample holder has very low thermal conductivity, the actual contact area between the cooling plate and the sample holder sidepieces is very small in practice. Due to the poor thermal contact, this design increased sample preparation time from approximately 3 hours (for the other systems) to approximately 6 hours [7]. The typical time required to cool down the dipstick after sample degas is around 2 hours.

Another problem associated with the design is that the two sides of the sample holder will be electrically shorted through the copper cooling plate while it is being cooled. Thus, unlike the other systems, it is impossible to flash the sample while keeping the dipstick at lower temperature.

3.1.2 The Updated Design

Due to the problems discussed above, it seems logical for an updated design to shorten the sample preparation time and to enable sample flash while cooling the dipstick. The idea is to build on the existing cooling plate with the nitrogen lines, and to increase the contact area between the sample holder/dipstick and the cooling plate.

As a first attempt, the concept is to design a copper "clip" structure which will sit on top of the existing cooling plate and wrap around the sidepieces of the sample holder for a bigger thermal contact area (illustrated in Figure 3.2). The clip will be in the "open" position when not in use (Figure 3.2 (a)) and can be closed when the sample is pressed down to ensure a good thermal contact (Figure 3.2 (b)). However, in practice the sample holder is sandwiched between rollers while it is on the dipstick. Thus, there is no flat surface for the clips to clamp.

The updated design is to add an attachment to the lower part of the dipstick to create a flat surface for the clips to clamp (Figure 3.3). In this case, the cooling plate will be in direct contact with the dipstick. To ensure dipstick cooling while flashing the sample, a sheet of Shapal-M is added between the existing cooling plate and the clip structure. Shapal-M is electrically insulating, thermally conductive, and machinable, which makes it an ideal material for this application. As illustrated in Figure 3.3, the clips are opened by a pair of beryllium-copper foils, clamped to the top of the clip and the side of the Shapal-M. Beryllium-copper is chosen because of its high mechanical strength and thermal conductivity. The design looks promising; however, the path of the LTM will be blocked when the cooling plate is put inside the preparation chamber, which makes sample transfer to the STM chamber impossible.

To solve this problem, the existing cooling plate is brought outward from the center of the preparation chamber, the "legs" under the cooling plate are lowered, and a "step" piece is added to link the two together with the Shapal-M layer sitting on top of the "step"

(Figure 3.4). Also, the Shapal-M layer and the clips are made rotatable with respect to the "step." In this way, the LTM can pass through when the longer edge of the Shapal-M is perpendicular to the LTM (Figure 3.4 (b)), and the sample can be seen from the viewing window when the Shapal-M is rotated 90° (Figure 3.4 (a)).

Figure 3.5 illustrates the entire design as it is implemented into the preparation chamber. It is shown at an angle from the direct front view to give a better picture of the structures inside. Detailed dimensions and specifications of each part described above are included in the schematics in the Appendix.

3.1.3 The Design Outcome and Result

Figures 3.6 and 3.7 show the actual cooling plate parts fabricated by the ECE machine shop. Each part is cleaned using standard cleaning procedures, which will be outlined in the following paragraphs.

Copper parts are first sonicated in Formula 409 Degreaser for five minutes to remove any oil left from the machining. After an overflow rinse in deionized water (DI water), they are sonicated in acetone and IPA, each for 5 minutes, to remove organic impurities. Acid etching is followed by dipping the copper parts in 1:3 HNO_3/DI for 20 seconds. Finally, the parts are cleaned again in acetone and IPA for 5 minutes each.

Critical copper parts (e.g. the "clips," "step," and dipstick attachments) are electro-polished, which replaces the acid etching step in the standard copper cleaning procedure. Compared with standard acid cleaning, electro-polished surfaces will be smoother, and thus will provide better thermal contact. Electro-polishing is carried out in a liquid electrolyte with phosphoric acid being its main constituent. A lead plate is used as the cathode while the copper parts are connected to the anode. The electrolyte is heated to 75-85 °F, while keeping a potential bias of 4-5 V between the electrodes. The electro-polishing time is between 5 and 8 min. This step will produce a shiny surface on the copper parts, which ensures good thermal contact.

The cleaning of the stainless steel parts (e.g. screws) is almost the same as that of copper, except that we replace the acid etch in nitric acid with dipping the parts in 1:1 HCl/DI for 10-12 seconds and 1:1 H_3PO_4/DI for 20 seconds. The cleaned stainless steel screws are then UHV compatible.

After the cooling plate is assembled and installed in the preparation chamber, tests are carried out to investigate the cooling efficiency. The dipstick temperature after sample degas is tabulated and compared with the data before the cooling plate update. The results are plotted in Figure 3.8. From the graph we can see that the time taken to cool the dipstick to below 40 °C has been significantly shortened from ~110 min to around 50 minutes. Though it still takes a longer time than the internal nitrogen pipe on the other system, the updated cooling plate is much more effective than it used to be, and shortens the sample preparation time from ~ 6 hours [7] to ~ 3 hours.

3.2 Capillary Doser Installation

Another major system update is the installation of a capillary doser. This is to facilitate the experiment on nanometer-scale selective silicon growth, which will be carried out in this STM system. In this experiment, a disilane precursor is used for the growth of Si nano-features on the surface.

The dosing of disilane in the STM chamber is controlled by a leak valve, while the pressure is monitored by the ion gauge located at the bottom of the STM chamber. In the existing setup, the disilane is dosed to the entire STM chamber; i.e., the disilane pressure is the same everywhere in the chamber. During a typical Si growth experiment, a base pressure of 10^{-7} torr is reached in the STM chamber, which is four orders of magnitude higher than the usual base pressure in the UHV STM chamber. Furthermore, it usually takes a few hours before the pressure can recover. Thus, it will be helpful to introduce a way to lower the dosing pressure while being able to carry out the experiment in the same manner.

The solution is to install a capillary doser, a small tube through which the precursor gas can pass directly to the sample/tip junction during the experiment (Figure 3.9). This small tubing concentrates the precursor gas to a small region, and as a result, the local precursor pressure can be orders of magnitude higher than the base pressure in the STM chamber.

The position of the doser can be adjusted by the doser manipulator located outside the chamber (Figure 3.10). The manipulator is able to control the doser position in the x-, y- and z-direction independently. During the experiment, the doser is moved to the tip-

sample junction; otherwise, it is located beside the scanner so that it is not blocking the path for tip transfer. Figure 3.11 shows the capillary doser inside the STM chamber.

With the capillary doser, experiments can be carried out with a chamber base pressure much lower than the current pressure used. A background pressure of 5 x 10^{-9} torr is capable of producing a local pressure of 1 x 10^{-5} torr in another system using a similar doser [8]. It is believed that the base pressure during the disilane experiment can be lowered by two orders of magnitude with the capillary doser installed.

3.3 System Operation

After the system updates, the STM system is brought back to UHV by a bakeout at 150 °C for five days. The base pressure in the STM and preparation chamber is in the range of 10^{-10} torr after the bake.

The operation of the system is tested by scanning a Si (100) surface prepared by the procedures outlined in Chapter 2. A flat silicon surface with dimer row directions perpendicular to each other in adjacent terraces can be seen in Figure 3.12. The system is now ready for further experiments on nanometer-scale silicon selective growth using disilane precursor.

3.4 Figures

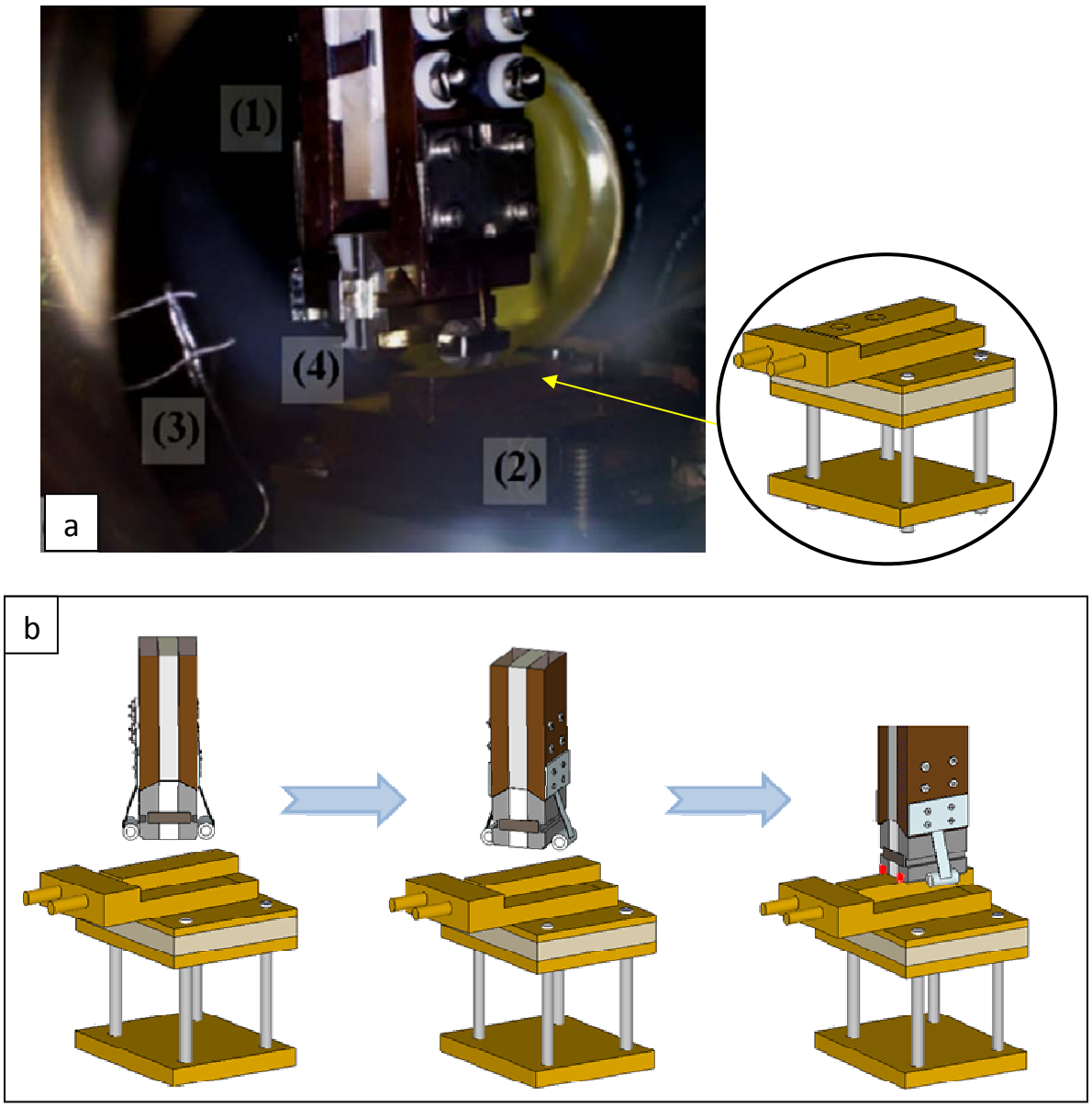

Figure 3.1: The original cooling plate design in the preparation chamber. **(a)** Photo of the original cooling plate. (1) Dipstick, (2) original cooling plate, (3) hydrogen cracking filament, (4) sample holder. Inset: diagram illustrating the original cooling plate supported by the "legs." **(b)** Diagram illustrating the working of original cooling plate. The dipstick is turned 45° clockwise from the position where the sample is facing the front, and pressed down to the cooling plate. The thermal contact area between the cooling plate and the sample holder is highlighted in red.

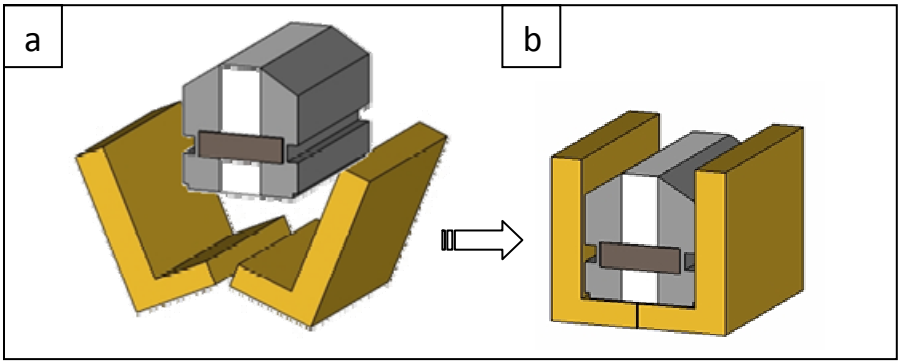

Figure 3.2: The first design idea. To increase the thermal contact area, a copper "clip" structure is designed to wrap around the side and bottom of the sample holder. **(a)** "Clips" in the "open" position when the cooling plate is not in use. **(b)** "closed" position during sample cooling.

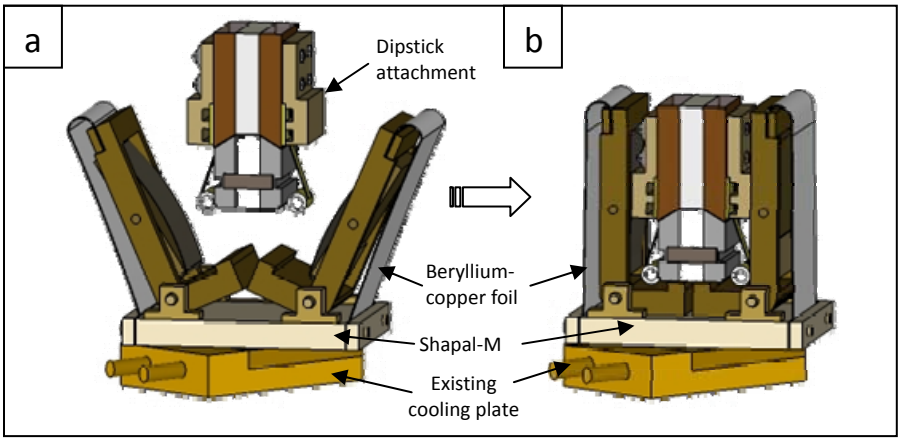

Figure 3.3: The updated design. Attachments are added to the dipstick to provide a flat surface for the "clips" to clamp. A Shapal-M sheet is used as the base of the copper "clips" for its electrical insulation and thermal conductivity. **(a)** The "clips" are kept in the "open" position by the Be-Cu foils, clamped to the side of the Shapal-M and the top of the clips. **(b)** The "clips" are closed by pressing the sample holder down.

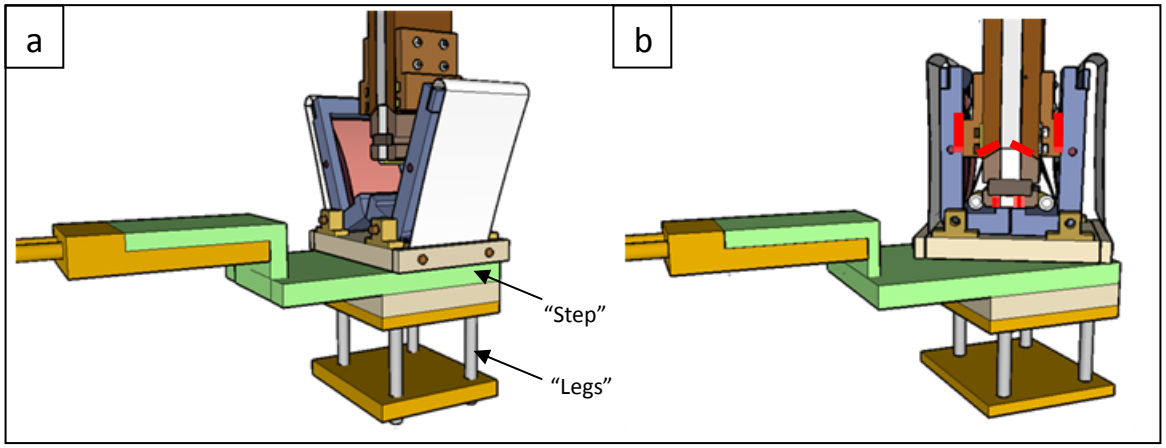

Figure 3.4: The final design. The "legs" are lowered to make room for the LTM to pass. The existing cooling plate and the Shapal-M piece are linked by a copper "step." The Shapal-M base is rotatable around its center. **(a)** Position of the cooling plate for sample transfer. **(b)** Position of the cooling plate during sample preparation. The two sides of the sample holder are electrically insulated and current can be passed through to flash the sample. The thermal contact area between the cooling plate and the sample holder is highlighted in red.

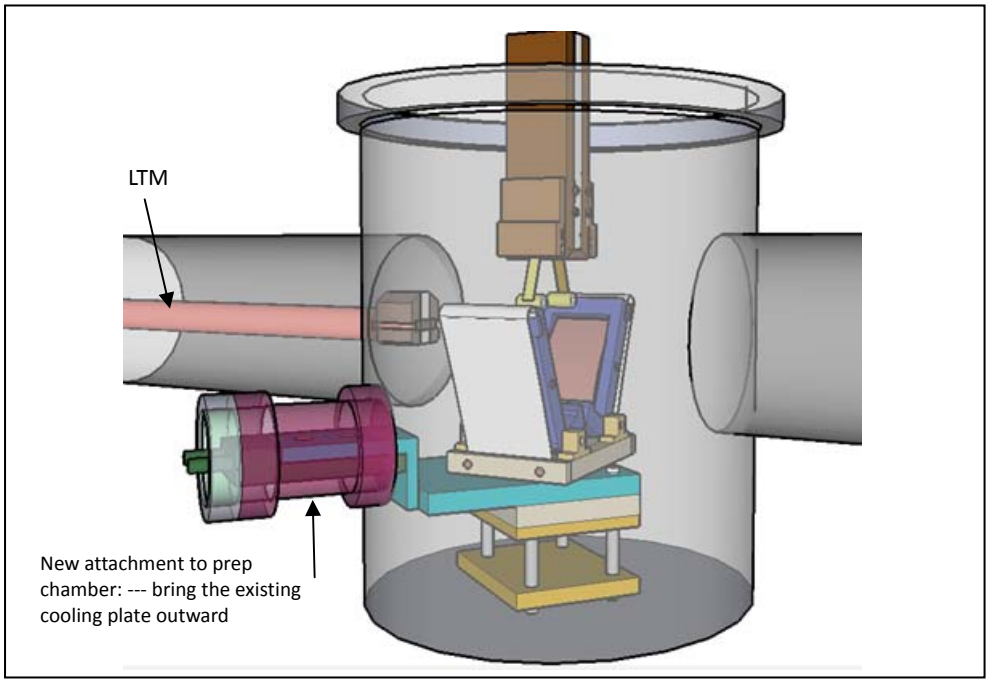

Figure 3.5: Final design of the cooling plate as placed in the preparation chamber.

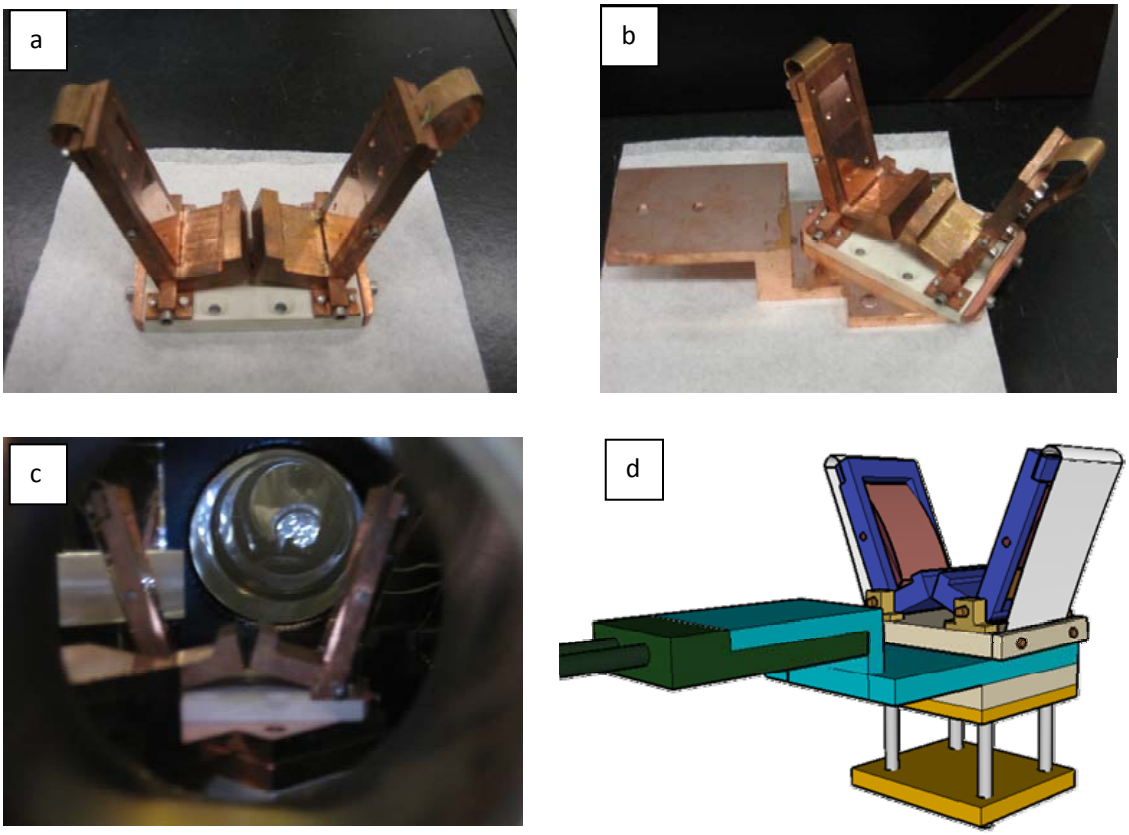

Figure 3.6: Cooling plate assembly on the bench and in the chamber. **(a)** Shapal-M base with copper "clips" and Be-Cu foils that keep the "clips" open. **(b)** Shapal-M base rotatable on the copper step that is used to connect to the existing cooling plate. **(c)** Cooling plate assembly inside the preparation chamber. **(d)** Diagram illustrating the cooling plate design.

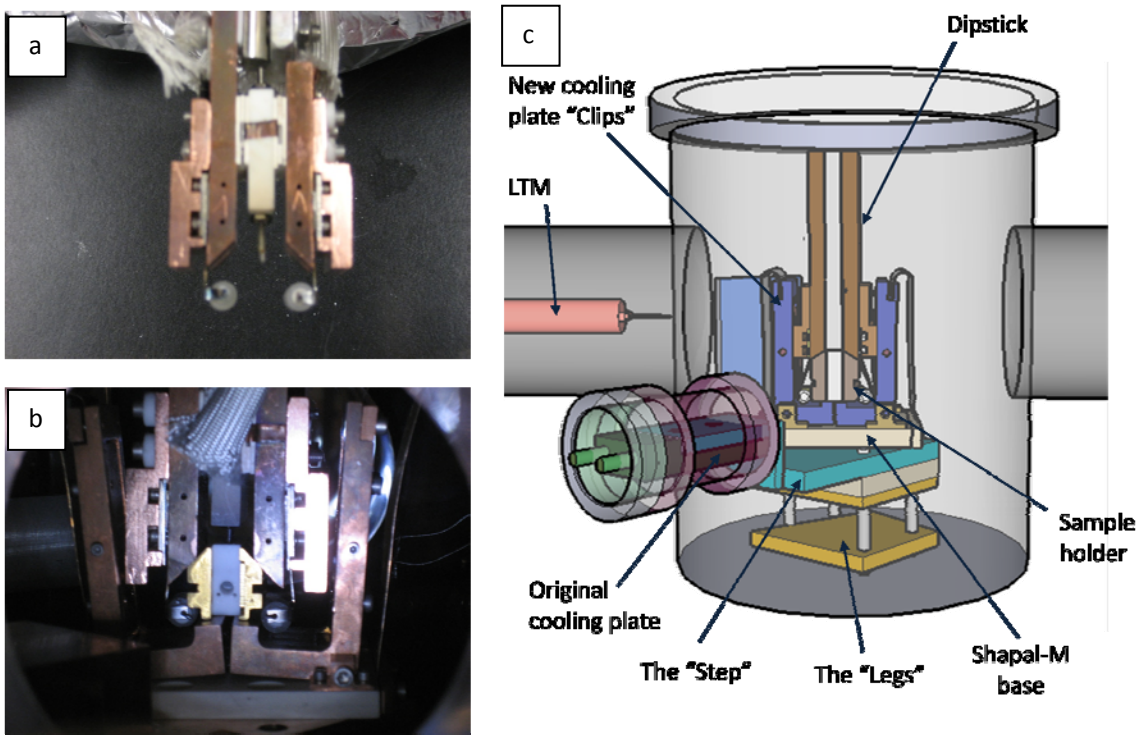

Figure 3.7: Dipstick attachment after it is installed. **(a**) On the bench, **(b)** inside the preparation chamber with the cooling plate and a sample holder, **(c)** schematic illustration for comparison.

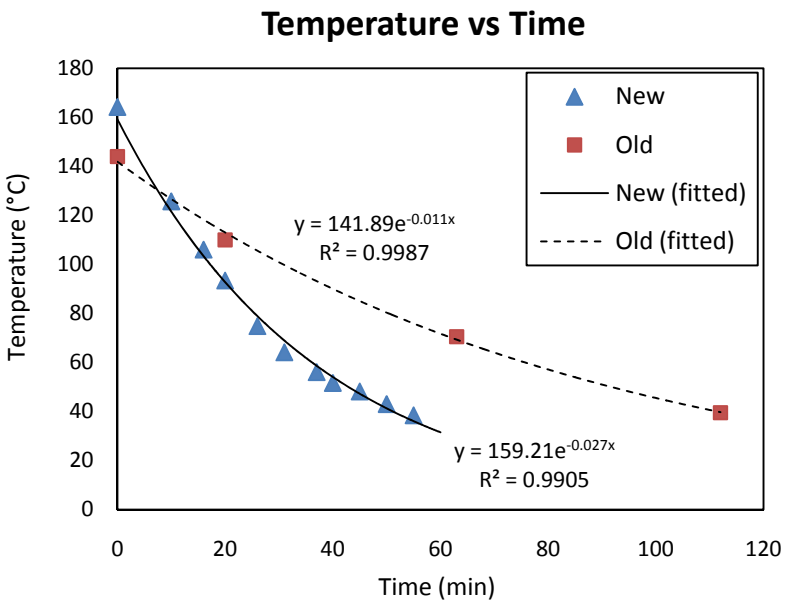

Figure 3.8: Comparison of cooling speed before and after updating the cooling plate.

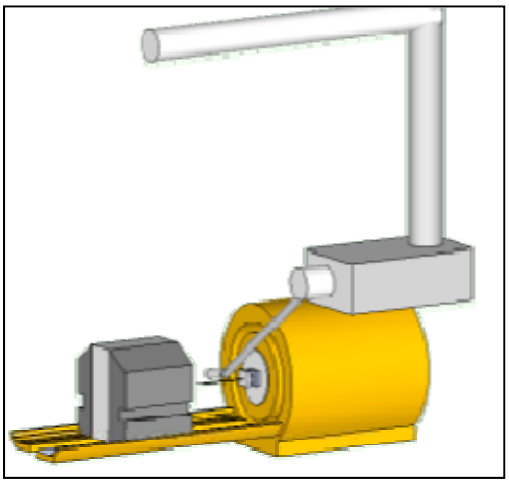

Figure 3.9: Diagram illustrating the position of capillary doser inside the STM chamber. The doser passes the precursor gas directly to the tip-sample junction.

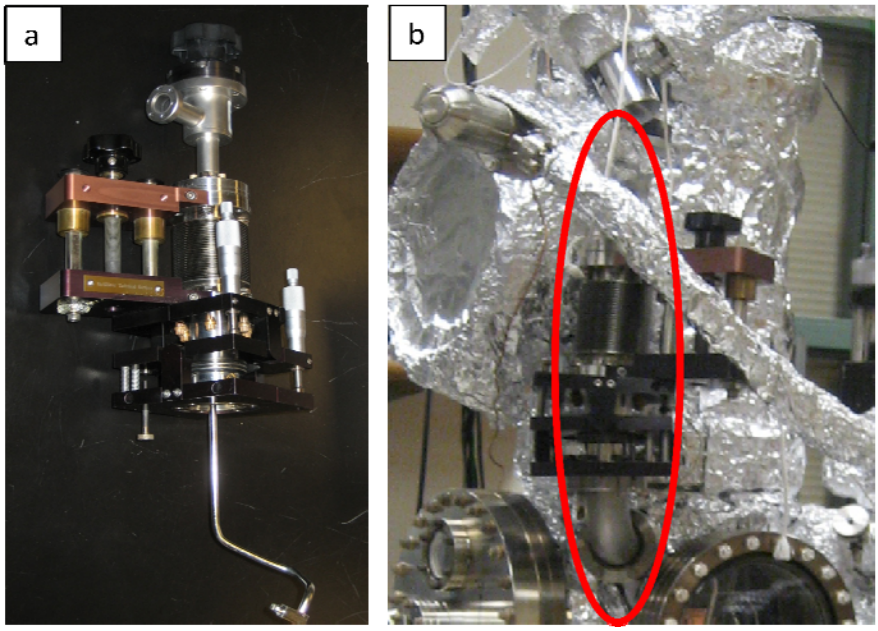

Figure 3.10: Capillary doser manipulator assembly. The x-, y-, z-position of the doser can be adjusted by the manipulator independently. **(a)** Doser manipulator on the bench with the doser attached to the end of the tubing. **(b)** Capillary doser installed to the system. The manipulator is circled in red.

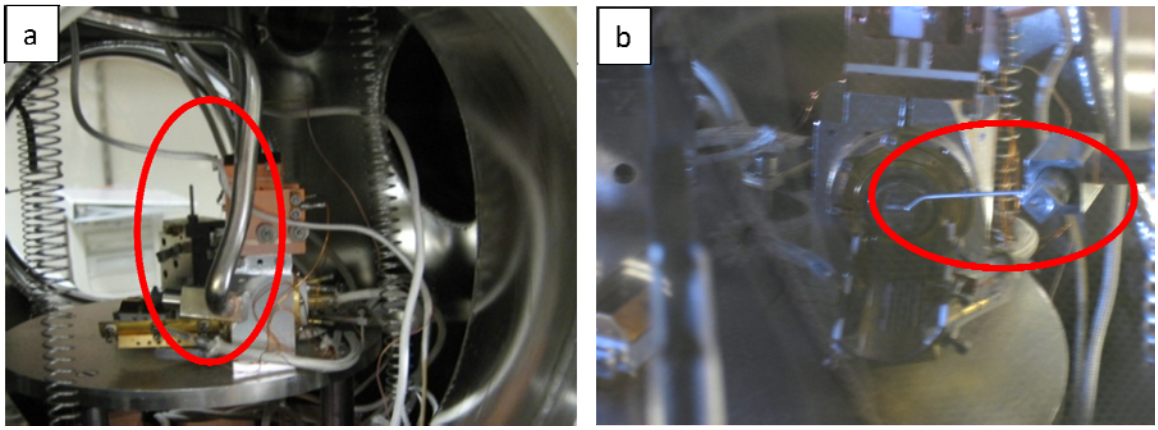

Figure 3.11: Capillary doser inside the STM chamber. The doser is placed at the tip/sample junction. **(a)** Side view, **(b)** front view, where the tip is facing the camera. (Sample is removed for photography purposes.)

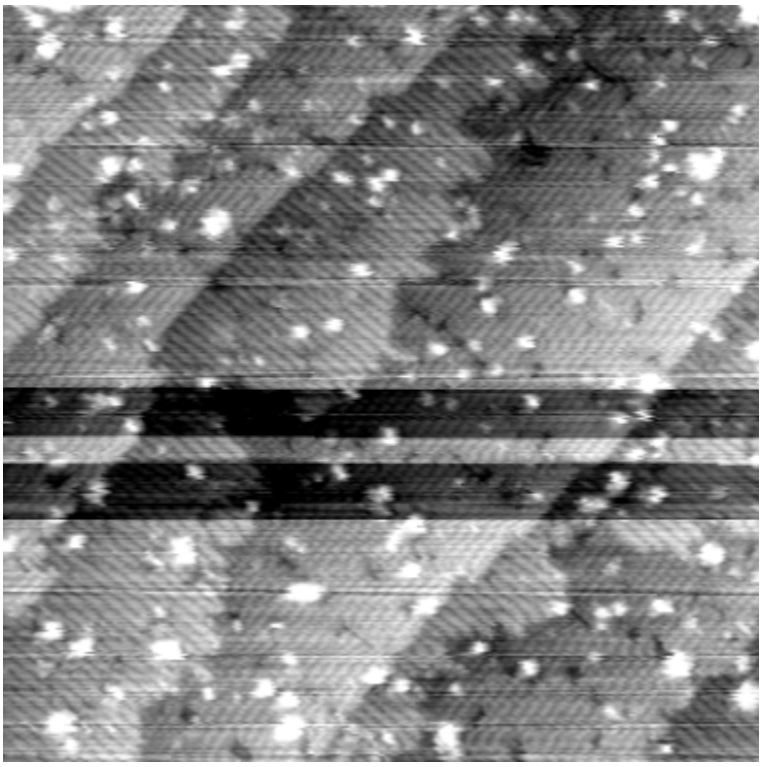

Figure 3.12: STM image acquired after the system update. Hydrogen-passivated Si (100)-2x1 surface with dimer row resolution can be seen. Image size: 500 x 500 Å; scan condition: -2 V, 0.1 nA.

Chapter 4: Conclusions

In this thesis, two major modifications to an ultra-high-vacuum scanning tunneling microscope system have been described: an update to the cooling plate structure for more effective cooling of the dipstick and sample holder, and the installation of a capillary doser for concentrating the precursor gas to the tip-sample junction during silicon nanostructure growth. The updated cooling plate is able to shorten the total sample preparation time from 6 hours to 3 hours. The capillary doser lowers the base pressure during the silicon growth experiment by two orders of magnitude. The system operation after the system modification was tested. STM images of hydrogen-passivated Si (100)-2x1 surface were acquired, and flat Si surfaces with dimer row resolution were seen.

The STM system is now ready for subsequent silicon nanostructure growth experiments. These experiments will use the monohydride layer on a hydrogen-passivated silicon surface as a lithographic mask, where a pattern can be defined by selectively desorbing the H atoms using the STM tip. The silicon growth is achieved by dosing the disilane gas. This method is proven to produce silicon structures on the nanometer scale. Future work will include investigating the effects of different parameters on silicon growth and achieving selective silicon epitaxial growth on the nanometer scale.

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Appendix: Cooling Plate Design Specifications

Schematics of the cooling plate parts described in Chapter 3 are provided in Figure A.1 to Figure A.6. These are the schematics given to the ECE machine shop. Detailed dimensions and specifications of each part as well as a 3D illustration of the part are included. Also, a brief description of each part is provided to discuss its function and structure.

A.1 Copper "Step"

The copper "step" (Figure A.1) is the connection between the original cooling plate and the new design. The purpose of this part is to lower the new cooling plate assembly by 0.5 inch so that there is enough space for sample transfer. Nitrogen gas is passed through the original cooling plate to keep it cool, and the heat from the dipstick is transferred through the "step" to be dissipated from the original cooling plate.

Two 6-32 countersunk screws are used to secure the upper "step" to the top of the original cooling plate, while the lower step is resting on the supporting "legs." A tap hole is drilled at the center of the lower step, where a 4-40 countersunk screw is used as the axle for the Shapal-M piece to rotate.

A.2 Shapal-M Base

The Shapal-M base (Figure A.2) sits on the lower step and supports the "clips" structure that will come in contact with the bottom of the dipstick attachment during cooling. Shapal-M is used because of its good thermal conductivity as well as its electrical insulation.

Four pairs of 0-80 tap holes are drilled on the Shapal-M to secure 4 holders (Figure A.5). The holders are used to hold the two axles around which the "clips" rotate. Also, one 4-40 countersunk hole is drilled in the center of the Shapal-M to hold the axle around which it rotates. Furthermore, two sidepieces are secured at the side of the Shapal-M base to hold the Be-Cu pieces that keep the clips open (Figure A.5).

A.3 Copper "Clips"

The copper clips (Figure A.3) are made of copper for its excellent thermal conduc-

tivity. The bottom corner of the clip is rounded so that it can rotate around the axle. The axle is a 1.5 inch long 2-56 screw which is held in place by two holders described in the previous section. The height of the clip is chosen to ensure effective cooling of both the sample holder and tip heater (after tip degas). The center of the clip is made hollow so that another copper piece (Figure A.4) can be fitted inside and rotates about its center. This adds an additional degree of freedom to the clips and ensures a better thermal contact between the clip and the dipstick attachment. Another two copper pieces are used to secure the Be-Cu piece to the top of the clips (Figure A.5).

A.4 Dipstick Attachments

The copper dipstick attachments (Figure A.6) are installed to the bottom of the dipstick to provide a flat surface for thermal contact. The shape of the attachment is designed to be fitted onto the dipstick and to make use of the existing drilled holes on the dipstick. The attachment is secured to the dipstick by four 2-56 screws that run through both sides of the dipstick. MACOR washers are used at one end of the screws to prevent the screws from shorting the two sides of the dipstick. Moreover, the attachments are designed in a step-shape. The step height is designed to be larger than the combined height of the screw head and washer, so that the screw head will not be in the way when the “clips” come in contact with the dipstick attachments.

A.5 Figures

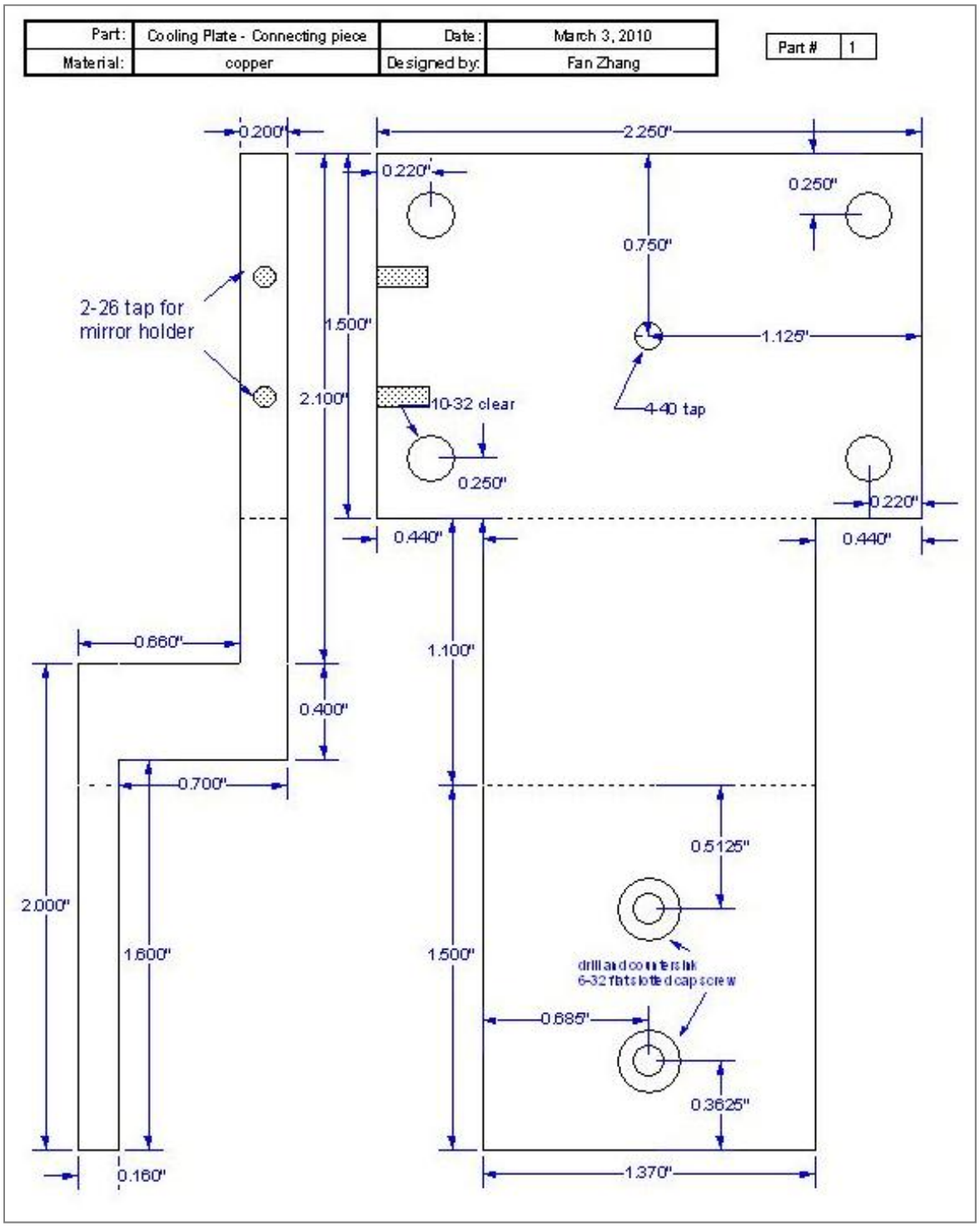
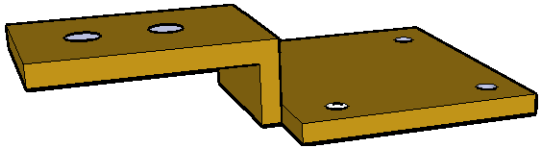


Figure A.1: Design specification of the "step".



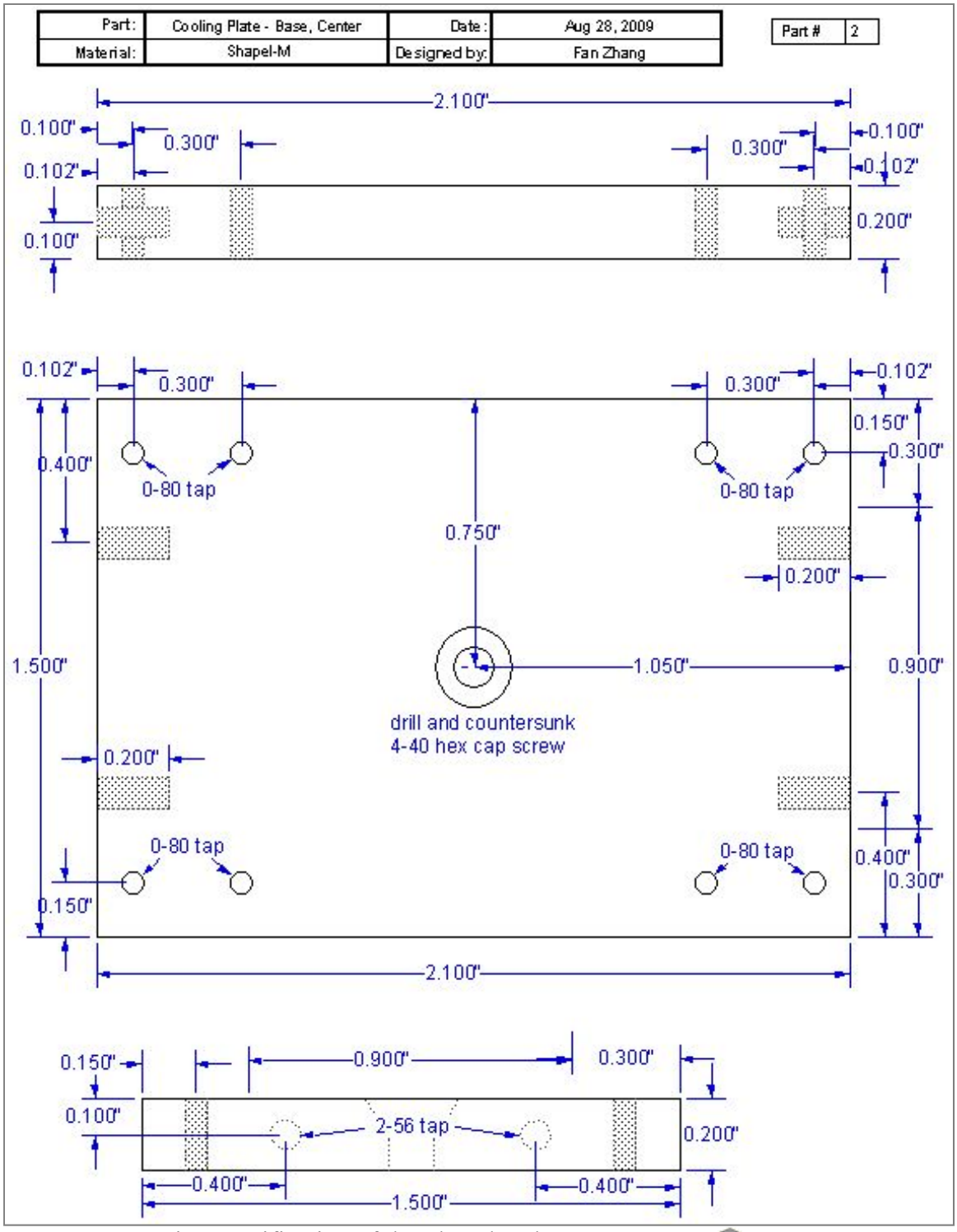
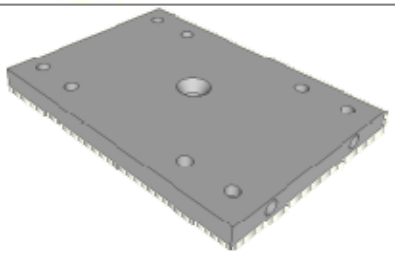


Figure A.2: Design specification of the Shapal-M base.



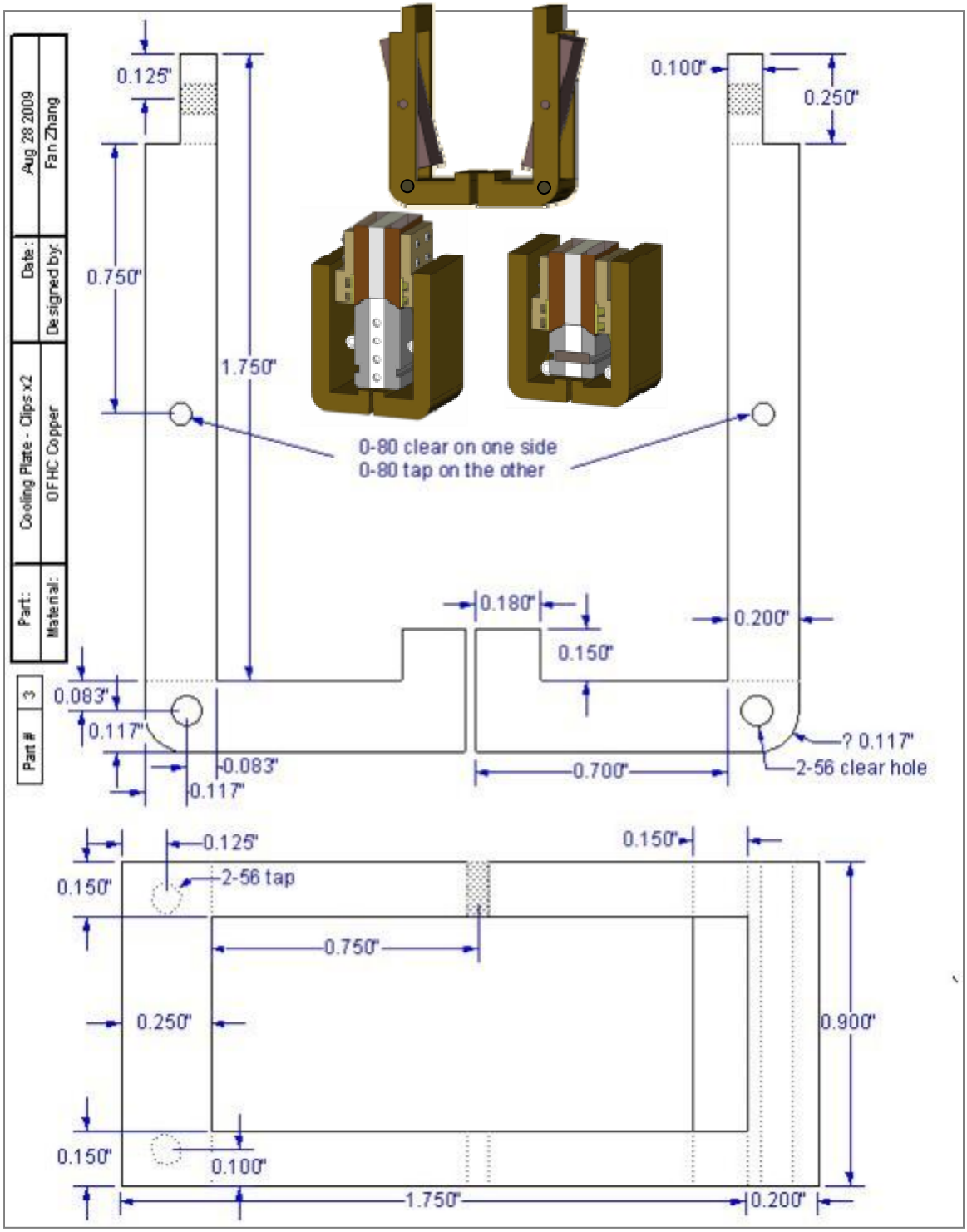


Figure A.3: Design specification of the "clips". The center is made hollow and another copper piece is fitted in the center. The center piece is made rotatable about the central axis to add another degree of freedom.

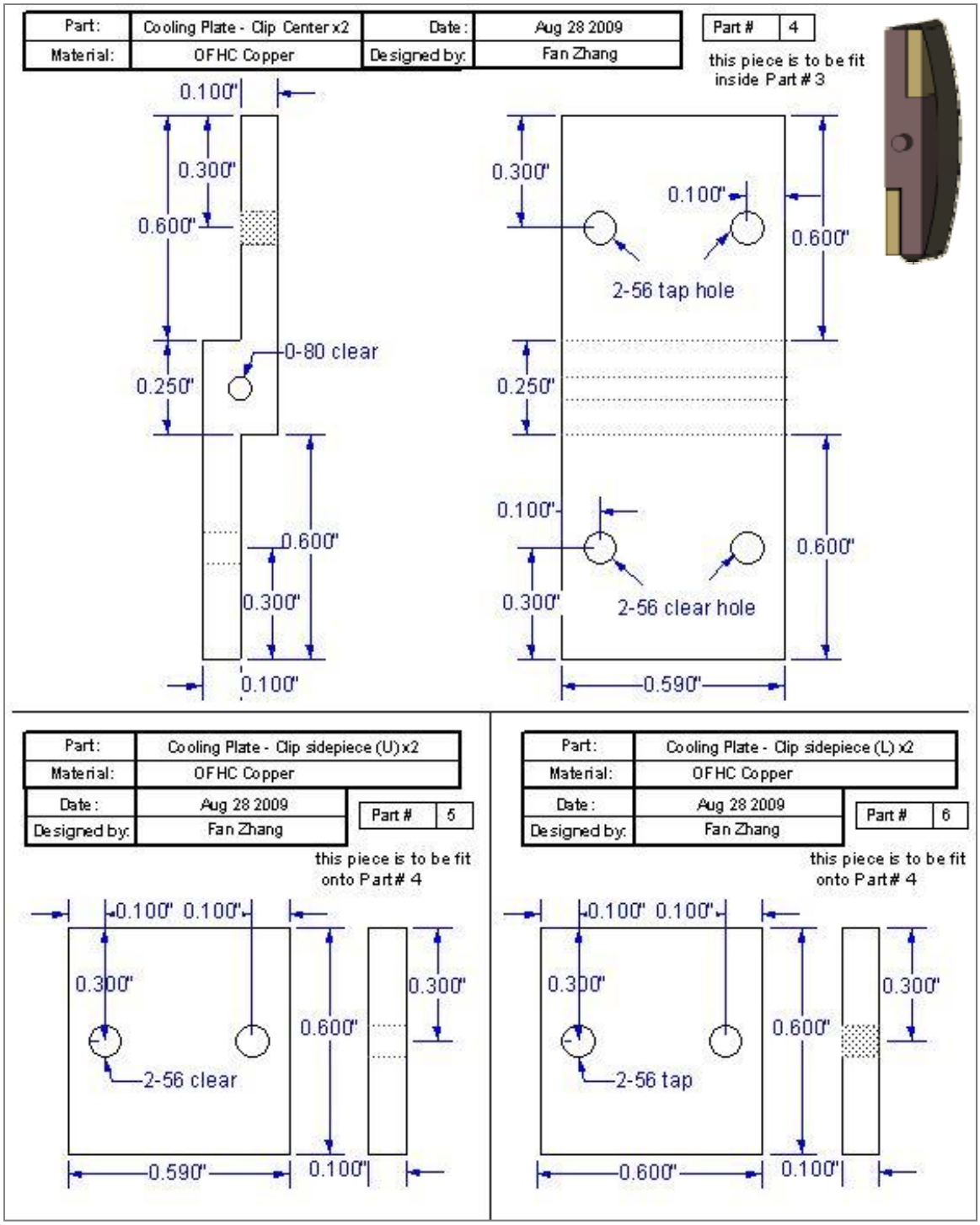


Figure A.4: Design specification of the center piece in the cooling plate clip.

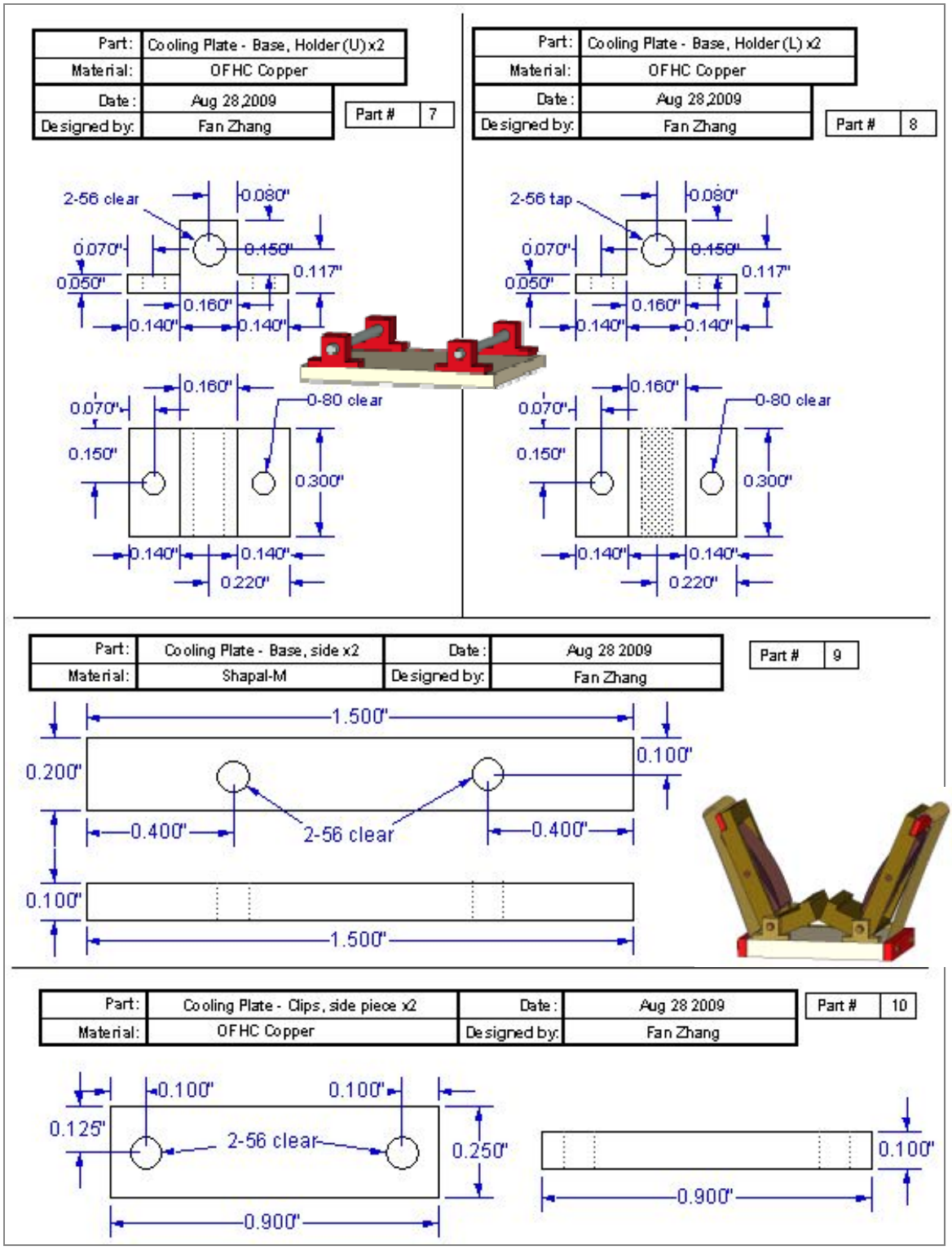


Figure A.5: Design specification of the sidepieces. Part #7 & #8: holders of the axles which hold the clips. Part #9: plates to clamp the Be-Cu foils to the sides of the Shapal-M. Part #10: plates to clamp the Be-Cu foils to the top of the clips.

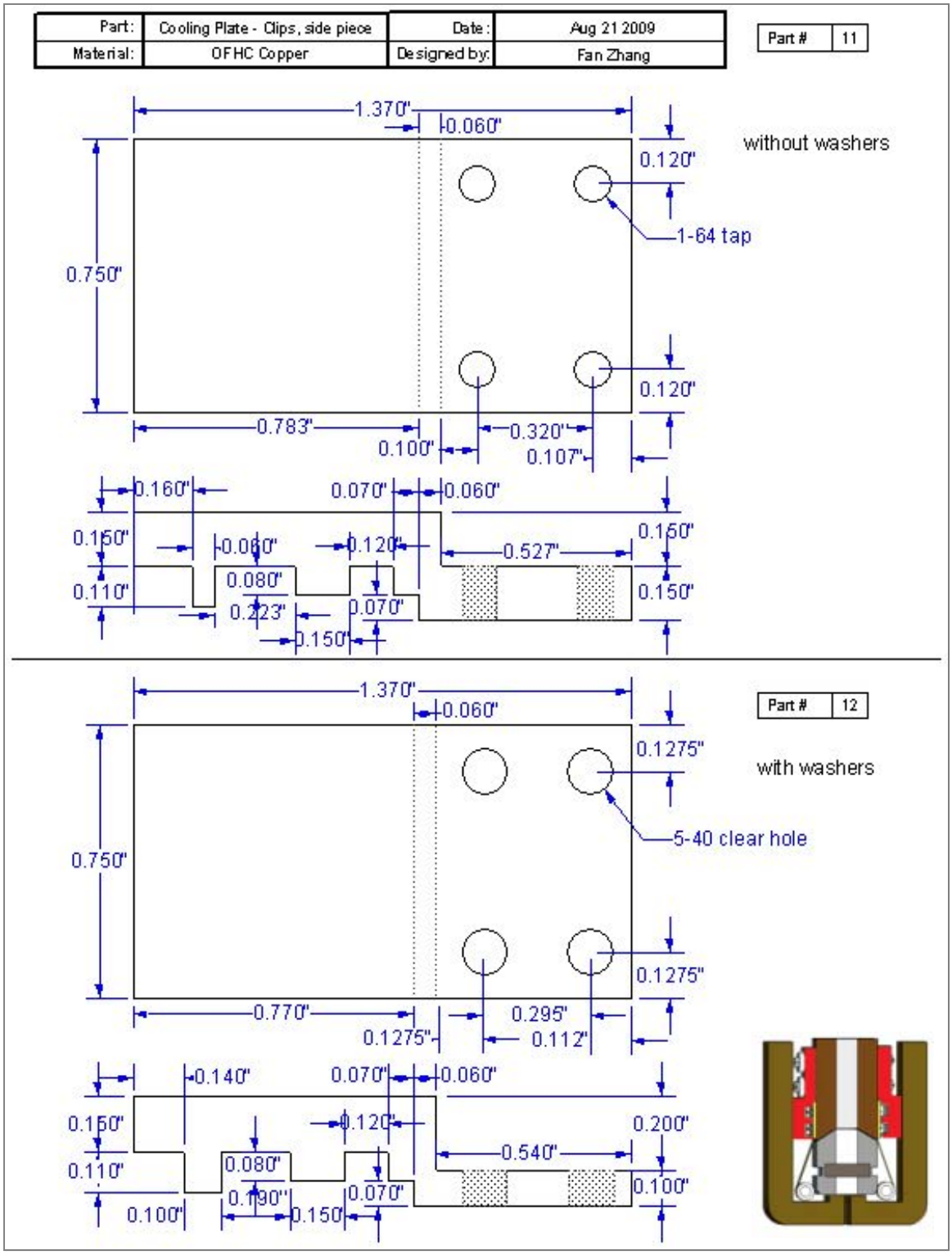


Figure A.6: Design specification of the dipstick attachments.